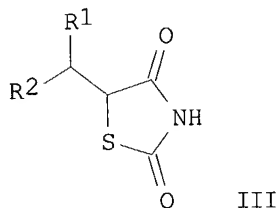
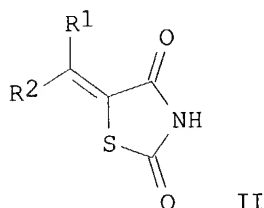


10/636,155

ACCESSION NUMBER: 134:100561 CASREACT
TITLE: Method for preparation of .alpha.-mercapto carboxylic acid
INVENTOR(S): Takahashi, Hiroshi; Ishii, Seichi; Yoshida, Kei; Tomiya, Kanji; Kodaka, Kenji
PATENT ASSIGNEE(S): Mitsui Chemical Industry Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 9 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2001011042	A2	20010116	JP 1999-187566	19990701

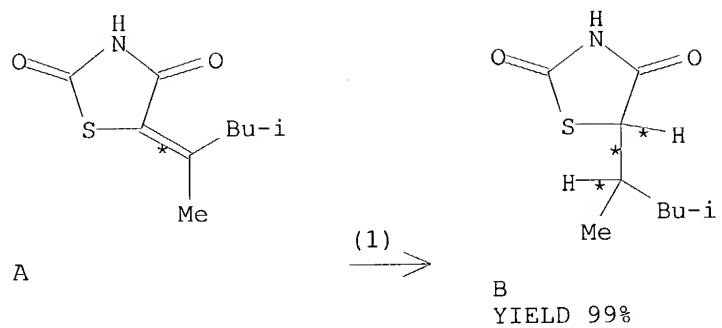
PRIORITY APPLN. INFO.: JP 1999-187566 19990701
OTHER SOURCE(S): MARPAT 134:100561
GI



AB .alpha.-Mercapto carboxylic acids represented by formula $R_1R_2CHCH(SH)CO_2H$ [I; $R_1, R_2 = C1-6$ alkyl or $R_1R_2C =$ (un)substituted C5-8 cycloalkane] are prepd. by condensation of thiazolidine-2,4-dione with R_1R_2CO ($R_1, R_2 =$ same as above) with $NH_3(g)$, primary alkylamine, or amine carbonate, bicarbonate, or acetate salts to obtain 5-alkylidenethiazolidine-2,4-diones (II; $R_1, R_2 =$ same as above) and catalytic hydrogenation of II to 5-alkylthiazolidine-2,4-diones (III; $R_1, R_2 =$ same as above) followed by hydrolysis to I. I are useful as intermediates for drugs or agrochems., e.g. fungicidal 2-alkyl-3-aminothiophenes. Thus, Me iso-Bu ketone was condensed with thiazolidine-2,4-dione in the presence of 0.82 g ammonium carbonate and 7.20 g $MgSO_4$ at 90.degree. for 11 h to give 79.8% 5-(1,3-dimethylbutylidene)thiazolidine-2,4-dione (98.5% purity) which was hydrogenated over 5% Pd-C in AcOH at 40 kg/cm² and 80.degree. for 6 h to give 99% 5-(1,3-dimethylbutyl)thiazolidine-2,4-dione. The latter compd. (15.0 g) was mixed with 93 mL H_2O , followed by blowing N, which was passed through blue silica gel, into the mixt. for 1 h, and 14.9 g NaOH was added in one portion and heated at 80.degree. for 8 h, cooled to room temp., and extd. with Et₂O. The aq. layer was adjusted to pH 1.0 with concd. HCl and extd. twice with Et₂O and the combined ext. was dried over $MgSO_4$, filtered, and concd. to give 81% 3,5-dimethyl-2-mercaptohexanoic acid.

RX(1) OF 6 ...A ==> B...

10/636,155

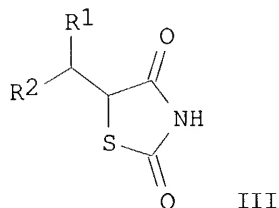
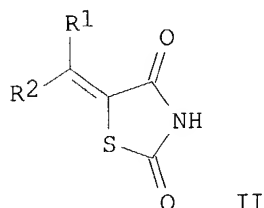


RX(1) RCT A **98959-96-7**
 RGT C 1333-74-0 H2
 PRO B **320350-17-2**
 CAT 7440-05-3 Pd
 SOL 64-19-7 AcOH

10/636,155

ACCESSION NUMBER: 134:100561 CASREACT
TITLE: Method for preparation of .alpha.-mercapto carboxylic acid
INVENTOR(S): Takahashi, Hiroshi; Ishii, Seiichi; Yoshida, Kei; Tomiya, Kanji; Kodaka, Kenji
PATENT ASSIGNEE(S): Mitsui Chemical Industry Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 9 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

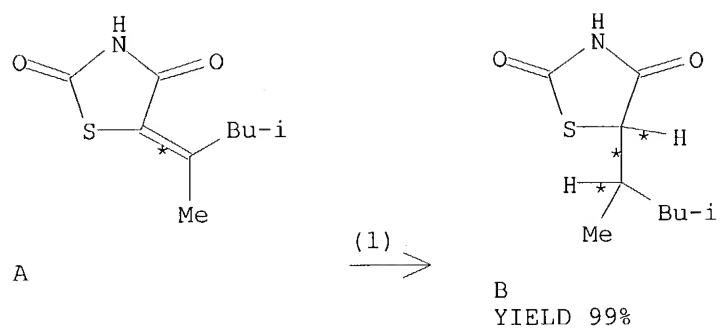
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2001011042	A2	20010116	JP 1999-187566	19990701
PRIORITY APPLN. INFO.:			JP 1999-187566	19990701
OTHER SOURCE(S):	MARPAT 134:100561			
GI				



AB .alpha.-Mercapto carboxylic acids represented by formula $R_1R_2CHCH(SH)CO_2H$ [I; $R_1, R_2 = C_1-6$ alkyl or $R_1R_2C = (un)substituted C_5-8$ cycloalkane] are prepd. by condensation of thiazolidine-2,4-dione with R_1R_2CO ($R_1, R_2 =$ same as above) with $NH_3(g)$, primary alkylamine, or amine carbonate, bicarbonate, or acetate salts to obtain 5-alkylidenethiazolidine-2,4-diones (II; $R_1, R_2 =$ same as above) and catalytic hydrogenation of II to 5-alkylthiazolidine-2,4-diones (III; $R_1, R_2 =$ same as above) followed by hydrolysis to I. I are useful as intermediates for drugs or agrochems., e.g. fungicidal 2-alkyl-3-aminothiophenes. Thus, Me iso-Bu ketone was condensed with thiazolidine-2,4-dione in the presence of 0.82 g ammonium carbonate and 7.20 g $MgSO_4$ at 90.degree. for 11 h to give 79.8% 5-(1,3-dimethylbutylidene)thiazolidine-2,4-dione (98.5% purity) which was hydrogenated over 5% Pd-C in AcOH at 40 kg/cm² and 80.degree. for 6 h to give 99% 5-(1,3-dimethylbutyl)thiazolidine-2,4-dione. The latter compd. (15.0 g) was mixed with 93 mL H_2O , followed by blowing N , which was passed through blue silica gel, into the mixt. for 1 h, and 14.9 g NaOH was added in one portion and heated at 80.degree. for 8 h, cooled to room temp., and extd. with Et₂O. The aq. layer was adjusted to pH 1.0 with concd. HCl and extd. twice with Et₂O and the combined ext. was dried over $MgSO_4$, filtered, and concd. to give 81% 3,5-dimethyl-2-mercaptohexanoic acid.

RX(1) OF 6 ...A ==> B...

10/636,155



RX(1)	RCT	A	98959-96-7
	RGT	C	1333-74-0 H2
	PRO	B	320350-17-2
	CAT		7440-05-3 Pd
	SOL		64-19-7 AcOH